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## AERONAUTICAL MATERIALS LABORATORY

REPORT NO. NAEC AML 1636      DATE 13 MARCH 1963

### FINAL REPORT

DEVELOPMENT AND FORMULATION OF EXPERIMENTAL  
DIISOCYANATE-BASED LAMINATING RESINS

PROBLEM ASSIGNMENT NO. C 09 RMA 31-1 UNDER  
BUREAU OF NAVAL WEAPONS  
WEPTASK RRMA 03 017/200 1/ROO7 04 01

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APR 30 1963

NAVAL AIR ENGINEERING CENTER  
PHILADELPHIA 12, PENNSYLVANIA

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ABSTRACT

The successful synthesis of halogenated polyurethane resins for laminating is reported. The test results indicate that halogen groupings in a polyurethane resin, tend to increase the thermal stability of the resulting laminate and to increase the pot-life of the resin.

Future efforts will be directed toward improving stability above 300°F of the polyurethane structural laminate.

A patent pertaining to the technique of formulating polyurethane resins has been allowed to Mr. A. P. Bonanni. This patent will be issued soon.

## I. INTRODUCTION

A. This is the final report under problem assignment C 09 RMA 31-1, WEPTASK RRMA 03 017/200 1/RO07 04 01 which was authorized by reference (a). Early experimentation (references (b), (c), (d) and (e)) indicated that the formulation of a polyurethane resin suitable for preparing a reinforced laminate was feasible. Also, that 2-butene-1,4-diol when reacted with 2,4-tolylene-diisocyanate (TDI) produced the best physical properties in a polyurethane structural laminate. This study pertains to the reaction of halogenated diols with TDI, to increase the pot-life of the polyurethane resin, and to increase the thermal stability of the structural laminates. This work was performed between February 1960 and February 1963.

## II. SUMMARY OF RESULTS

A. Improved flexural modulus properties at elevated temperatures have been attained with the use of halogenated groupings in the polyurethane resins. The physical properties of these structural laminates could be improved with further experimentation.

B. The pot-life, a problem with un-halogenated diols, is extended to several days with the use of halogenated diols.

### III. CONCLUSIONS

A. The thermal stability of a structural laminate, increases with the use of halogenated diols. Further improvement can be made by varying the concentration of TDI and Macconate 200. Although the flexural properties do not appear to be significantly high, they must not be considered as the laminate's ultimate flexural strength. In order to determine their ultimate flexural strength, a study must be made to determine the temperature, pressure and time of lamination.

B. The pot-life of a polyurethane resin can be extended by modifying the chemical structure of the experimental resin using halogenated diols.

### IV. RECOMMENDATIONS

A. It is recommended that a new problem assignment be established to:

1. Determine optimum laminating parameters of a halogenated diol and a diisocyanate resin which will give superior physical properties in a structural laminate.
2. Ascertain if a peroxide will increase cross-linking in the unsaturated polymers.
3. Determine the molecular weight of a polyurethane resin.
4. Study the physical properties of structural laminates formulated with varying amounts of:
  - a. 2,4-tolylene-diisocyanate
  - b. 2-butene-1,4-diol
  - c. Halogenated diols
  - d. 3,3'-bitolylene-4,4'-diisocyanate and 3,3'-dimethyldiphenylmethane and the laminating parameters (temperature, pressure and time).

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## V. DESCRIPTION, METHODS, RESULTS

### A. Description

1. Materials used in this investigation were obtained from sources shown in Plate 1. All the materials reported herein are new to the investigation of polyurethane resins with the exception of TDI and 2-butene-1,4-diol.

### B. Method

1. The reactants used in the formulation of the polyurethane resins reported herein are TDI, Macconate 200 (3,3'-bitolylene-4,4'-diisocyanate, Macconate 300 (diphenylmethane-4,4'-diisocyanate), 2-butene-1,4-diol, HFPD, DEBD and tetrachlorobutane diol (TCDB).

2. A study of the melting points of halogenated polyurethane resins using the Koffler Hot Stage (Plate 2) was used to indicate the thermal stability of the resin. These softening points indicated that the physical properties of the structural laminate should be investigated at elevated temperatures. These results are reported in Plates 3 to 11 and graphically presented on Plates 12 to 19. Tests for the evaluation of melting points were performed in accordance with WADC Technical Report 56-399 under the title "Test Methods for Estimation of Thermal Behavior of Polymers."

3. All the structural laminates were prepared using 181 Volan A glass cloth. The laminates were cured at 30 psig and 300°F for 30 minutes and post cured at 158°F for 16 hours. Flexural strength and flexural moduli tests were utilized as a screening measure to compare the different polyurethane formulations; also, to make a comparison of the polyurethane resins at room temperature and elevated temperatures (200°F). The flexural properties were conducted in accordance with Test Method Standard No. 406, Plastics: Method of Testing.

### C. Results

1. An examination of the melting points, using the Koffler Hot Stage, indicates that as the molar concentration of TDI is increased and the alkene diol (2-butene-1,4-diol) is kept constant, the softening points of the resins increase to a maximum of 226°F with a molar construction of 1.2 moles of TDI. But when a halogenated diol is substituted for the alkene diol, the softening point increases steadily with the addition of more TDI. The softening points become more significant when polyurethane resins are synthesized using TDI, halogenated diols and Macconate 200. These resins do not soften, but begin to carbonize at 572°F. The results are tabulated and shown on Plate 2.

2. An examination of the flexural values at room temperature, (Plates 3, 5, 7, 9, and 11 to 19) and at 200°F after being exposed to 200°F for 1/2 hour (Plates 4, 6, 8, and 10 to 19) indicates that although some formulations show temperature stability, there are indications that the laminating parameters (curing temperature, pressure and time) used in this study are not the optimum for the laminates. On Plates 3, 11 and 12, the data show extremely low flexure values for a polyurethane resin using 1.0 mole of HFPD and 1.0 mole of TDI; but, with the increase of only a 0.10 of mole of TDI, a flexural value of 90,100 psi and flexural modulus of  $4.07 \times 10^6$  is obtained. When exposed to 200°F for 1/2 hour and tested at 200°F (Plates 4, 11 and 12) a loss of more than 70% of the flexural strength is seen and a loss of only 10% of the flexural modulus. On Plates 5, 13 and 14, using 1.0 mole of DBBD and 1.1 moles of TDI a flexural strength of 93,900 psi and a modulus of  $4.24 \times 10^6$  psi is obtained. When exposed to 200°F for 1/2 hour and tested at 200°F (Plates 6, 13 and 14) a loss of less than 3% is found in the flexural strength and a loss of approximately 5% in the flexural modulus. These values substantiate the theory that when halogenated diols are used, the thermal properties can be increased.

3. The pot-life of polyurethane resins is extended to several days when halogenated diols are used. In the formulation using HFPD and TDI, the resulting polyurethane resin remains a liquid for several days provided the resin is placed in an air-tight container free of moisture. The formulations using DBBD and TDI form a polyurethane resin which solidifies in 2 to 3 hours after synthesis but can be poured as a hot melt, provided the temperature of the resin is maintained between 190°F to 205°F. Formulating TDI with HFPD and DBBD and various concentrations of Nacconate 200 yielded resins having a pot-life of less than one hour. Formulating TDI with DBBD and Nacconate 300 yielded a resin which had a pot-life of approximately one-half hour. Once these resins are polymerized, they cannot be reheated to a melt.

REFERENCES

- (a) BUAER ltr Aer-AE-44/127 of Aug 1955
- (b) MAXSTA ltr XM-52-SP:dsf J2-5 (3517) of Aug 1955
- (c) Report No. NAMC AML AE 4420, Part I of 2 Mar 1956
- (d) Report No. NAMC AML AE 1071, (AE 4420, Part II) of 16 Oct 1958
- (e) Report No. NAMC AML 1131 (Project No. TED NAM AE 4420, Part III) of 22 Jun 1960

SOURCES OF MATERIALS

| <u>Compound</u>                   | <u>Source</u>   |
|-----------------------------------|---|
| 2,4-Tolylene-Diisocyanate         | E. I. duPont de Nemours & Co., Inc.<br>Wilmington 98, Delaware  |
| 3,3'-Bitolylene-4,4'-Diisocyanate | National Aniline Division<br>Allied Chemical & Dye Corporation<br>40 Rector Street<br>New York 6, New York            |
| Diphenylmethane-4,4'-Diisocyanate | Same  |
| 2-Butene-1,4-Diol                 | General Aniline & Film Corporation<br>Commercial Development Department<br>435 Hudson Street<br>New York 14, New York |
| Hexafluoropentane Diol            | Harker Chemical Corporation<br>Niagara Falls, New York  |
| Tetrachlorobutane Diol            | General Aniline & Film Corporation<br>Commercial Development Department<br>435 Hudson Street<br>New York 14, New York |
| Dibromobutene Diol                | Same  |

MELTING POINTS USING KOFFLER HOT STAGE (\*F)

| <u>Sample</u> | <u>Moles of</u>          |                            |             | <u>Softening<br/>Point</u>      | <u>Initial<br/>Flow</u> | <u>Complete<br/>Melt</u> |
|---------------|--------------------------|----------------------------|-------------|---------------------------------|-------------------------|--------------------------|
|               | <u>TDI<sup>(1)</sup></u> | <u>2B14D<sup>(2)</sup></u> | <u>---</u>  |                                 |                         |                          |
| 1             | 1.0                      | 1.0                        |             |                                 |                         |                          |
| 2             | 1.1                      | 1.0                        |             | 212                             | 230                     | 473                      |
| 3             | 1.2                      | 1.0                        |             | 248                             | 293                     | 426                      |
| 4             | 1.3                      | 1.0                        |             | 226                             | 248                     | 392                      |
|               | <u>TDI</u>               | <u>HFPD<sup>(3)</sup></u>  |             |                                 |                         |                          |
| 1             | 1.0                      | 1.0                        |             | ---                             | 381                     | 419                      |
| 2             | 1.1                      | 1.0                        |             | 241                             | 284                     | 340                      |
| 3             | 1.2                      | 1.0                        |             | 248                             | 401                     | 473                      |
| 4             | 1.3                      | 1.0                        |             | 365                             | 388                     | 410                      |
|               | <u>TDI</u>               | <u>DBBD<sup>(4)</sup></u>  |             |                                 |                         |                          |
| 1             | 1.0                      | 1.0                        |             | 203                             | 214                     | 221                      |
| 2             | 1.1                      | 1.0                        |             | 207                             | 234                     | 273                      |
| 3             | 1.2                      | 1.0                        |             | 248                             | 374                     | 410                      |
| 4             | 1.3                      | 1.0                        |             | ---                             | ---                     | ---                      |
|               | <u>TDI</u>               | <u>TCBD<sup>(5)</sup></u>  |             |                                 |                         |                          |
| 1             | 1.0                      | 1.0                        |             | 347                             | 392                     | 424                      |
|               | <u>TDI</u>               | <u>Naconate<br/>200</u>    | <u>HFPD</u> |                                 |                         |                          |
| 1             | 1.0                      | 0.5                        | 1.5         | 158                             | 169                     | 190                      |
| 2             | 0.75                     | 0.75                       | 1.5         | 255                             | 324                     | Charred at<br>572        |
| 3             | 0.50                     | 1.0                        | 1.5         | 243                             | 266                     | 302                      |
|               | <u>TDI</u>               | <u>Naconate<br/>200</u>    | <u>DBBD</u> |                                 |                         |                          |
| 1             | 1.0                      | 0.050                      | 1.5         | -----Sample Charred at 572----- |                         |                          |
| 2             | 0.75                     | 0.75                       | 1.5         | 262                             | 313                     | 360                      |
| 3             | 0.50                     | 1.0                        | 1.5         | 257                             | 293                     | Charred at<br>477        |

NOTES: (1) TDI - 2,4-Tolylene-Diisocyanate  
 (2) 2B14D - 2-Butene-1,4-Diol  
 (3) HFPD - Hexafluoropentane Diol  
 (4) DBBD - Dibromobutene Diol  
 (5) TCBD - Tetrachlorobutane Diol

FLEXURAL DATA FOR HEXAFLUOROPENTANE DIOL (HFPD)  
AND 2,4-TOLYLENE-DIISOCYANATE (TDI)

| Spec.<br>No. | Moles |      | Width<br>(in.) | Thick.<br>(in.) | Ultimate<br>Load<br>(lbs.) | Flexural<br>Strength<br>(psi) | E x 10 <sup>-6</sup> |       |      |
|--------------|-------|------|----------------|-----------------|----------------------------|-------------------------------|----------------------|-------|------|
|              | TDI   | HFPD |                |                 |                            |                               | Avg.                 | (psi) | Avg. |
| 1            | 1.0   | 1.0  | 0.498          | 0.124           | 56                         | 21,904                        | --No Results--       |       |      |
| 2            |       |      | 0.513          | 0.124           | 53                         | 20,126                        |                      |       |      |
| 3            |       |      | 0.502          | 0.125           | 54                         | 20,690                        |                      |       |      |
| 4            |       |      | 0.509          | 0.123           | 52                         | 20,312                        |                      |       |      |
| 5            |       |      | 0.516          | 0.124           | 52                         | 19,623                        | 20,500               |       |      |
| 1            | 1.1   | 1.0  | 0.500          | 0.088           | 115                        | 89,600                        |                      | 4.20  |      |
| 2            |       |      | 0.499          | 0.087           | 115                        | 91,000                        |                      | 4.17  |      |
| 3            |       |      | 0.497          | 0.090           | 120                        | 89,300                        |                      | 3.92  |      |
| 4            |       |      | 0.050          | 0.088           | 116                        | 90,400                        |                      | 4.11  |      |
| 5            |       |      | 0.498          | 0.089           | 118                        | 90,100                        | 90,100               | 3.97  | 4.07 |
| 1            | 1.2   | 1.0  | 0.515          | 0.089           | 116                        | 85,500                        |                      | 4.32  |      |
| 2            |       |      | 0.516          | 0.088           | 114                        | 86,200                        |                      | 4.48  |      |
| 3            |       |      | 0.528          | 0.089           | 123                        | 88,500                        |                      | 4.26  |      |
| 4            |       |      | 0.521          | 0.090           | 122                        | 86,700                        |                      | 4.14  |      |
| 5            |       |      | 0.517          | 0.090           | 118                        | 84,500                        | 86,300               | 4.29  | 4.30 |
| 1            | 1.4   | 1.0  | 0.530          | 0.103           | 19                         | 8,407                         | --No Results--       |       |      |
| 2            |       |      | 0.505          | 0.111           | 18                         | 8,696                         |                      |       |      |
| 3            |       |      | 0.510          | 0.110           | 18                         | 8,752                         |                      |       |      |
| 4            |       |      | 0.510          | 0.112           | 13                         | 6,113                         |                      |       |      |
| 5            |       |      | 0.509          | 0.111           | 13                         | 6,903                         | 7,800                |       |      |

FLEXURAL DATA FOR HEXAFLUOROPENTANE DIOL (HFPD)  
AND 2,4-TOLYLENE-DIISOCYANATE (TDI),  
TESTED AT 200°F AFTER EXPOSURE TO 200°F FOR 1/2 HOUR

| Spec.<br>No. | Moles |      | Width<br>(in.)                               | Thick.<br>(in.) | Ultimate<br>Load<br>(lbs.) | Flexural<br>Strength<br>(psi) | E x 10 <sup>-6</sup> |       |      |
|--------------|-------|------|--|-----------------|----------------------------|-------------------------------|----------------------|-------|------|
|              | TDI   | HFPD |  |                 |                            |                               | Avg.                 | (psi) | Avg. |
| 1            | 1.0   | 1.0  | -----Not Tested Because of Delamination----- |                 |                            |                               |                      |       |      |
| 2            |       |      |  |                 |                            |                               |                      |       |      |
| 3            |       |      |  |                 |                            |                               |                      |       |      |
| 4            |       |      |  |                 |                            |                               |                      |       |      |
| 5            |       |      |  |                 |                            |                               |                      |       |      |
| 1            | 1.1   | 1.0  | 0.502  | 0.087           | 32                         | 25,100                        |                      | ---   |      |
| 2            |       |      | 0.512  | 0.090           | 39                         | 28,200                        |                      | 3.43  |      |
| 3            |       |      | 0.495  | 0.089           | 35                         | 26,800                        |                      | 3.55  |      |
| 4            |       |      | 0.503  | 0.088           | 36                         | 27,900                        |                      | 3.59  |      |
| 5            |       |      | 0.500  | 0.089           | 34                         | 25,800                        | 26,800               | 3.43  | 3.50 |
| 1            | 1.2   | 1.0  | 0.530  | 0.090           | 52                         | 36,400                        |                      | 2.95  |      |
| 2            |       |      | 0.527  | 0.090           | 49                         | 34,400                        |                      | 3.35  |      |
| 3            |       |      | 0.512  | 0.088           | 43                         | 32,700                        |                      | 3.77  |      |
| 4            |       |      | 0.518  | 0.089           | 46                         | 33,700                        |                      | 3.70  |      |
| 5            |       |      | 0.519  | 0.088           | 44                         | 33,000                        | 34,100               | 3.87  | 3.49 |
| 1            | 1.4   | 1.0  | -----Not Tested Because of Delamination----- |                 |                            |                               |                      |       |      |
| 2            |       |      |  |                 |                            |                               |                      |       |      |
| 3            |       |      |  |                 |                            |                               |                      |       |      |
| 4            |       |      |  |                 |                            |                               |                      |       |      |
| 5            |       |      |  |                 |                            |                               |                      |       |      |

FLEXURAL DATA FOR DIEROMOBUTENE DIOL (DEBD) AND  
2,4-TOLYLENE-DIISOCYANATE (TDI) TESTED AT ROOM TEMPERATURE

| Spec.<br>No. | Moles      |             | Width<br>(in.) | Thick.<br>(in.) | Ultimate<br>Load<br>(lbs.) | Flexural<br>Strength<br>(psi) | E x 10 <sup>-6</sup> |              |             |
|--------------|------------|-------------|----------------|-----------------|----------------------------|-------------------------------|----------------------|--------------|-------------|
|              | <u>TDI</u> | <u>DEBD</u> |                |                 |                            |                               | <u>Avg.</u>          | <u>(psi)</u> | <u>Avg.</u> |
| 1            | 1.0        | 1.0         | 0.512          | 0.123           | 95                         | 59,200                        |                      | 3.95         |             |
| 2            |            |             | 0.501          | 0.123           | 95                         | 56,900                        |                      | 3.66         |             |
| 3            |            |             | 0.511          | 0.123           | 95                         | 58,000                        |                      | 3.75         |             |
| 4            |            |             | 0.497          | 0.123           | 98                         | 63,000                        |                      | 3.90         |             |
| 5            |            |             | 0.513          | 0.123           | 88                         | 54,800                        | 58,400               | 3.97         | 3.85        |
| 1            | 1.1        | 1.0         | 0.495          | 0.095           | 140                        | 94,200                        |                      | 4.23         |             |
| 2            |            |             | 0.520          | 0.098           | 154                        | 92,600                        |                      | 4.08         |             |
| 3            |            |             | 0.511          | 0.098           | 150                        | 91,600                        |                      | 4.23         |             |
| 4            |            |             | 0.495          | 0.094           | 139                        | 95,600                        |                      | 4.41         |             |
| 5            |            |             | 0.522          | 0.097           | 155                        | 94,700                        | 93,900               | 4.23         | 4.24        |
| 1            | 1.2        | 1.0         | 0.526          | 0.101           | 101                        | 56,400                        |                      | 3.75         |             |
| 2            |            |             | 0.506          | 0.096           | 93                         | 59,900                        |                      | 4.03         |             |
| 3            |            |             | 0.528          | 0.097           | 105                        | 63,500                        |                      | 4.08         |             |
| 4            |            |             | 0.519          | 0.100           | 90                         | 52,000                        |                      | 3.97         |             |
| 5            |            |             | 0.532          | 0.101           | 93                         | 51,400                        | 56,700               | 3.92         | 3.95        |
| 1            | 1.3        | 1.0         | 0.507          | 0.100           | 58                         | 34,800                        |                      | 3.30         |             |
| 2            |            |             | 0.525          | 0.096           | 61                         | 38,200                        |                      | 3.47         |             |
| 3            |            |             | 0.486          | 0.096           | 55                         | 36,900                        |                      | 3.95         |             |
| 4            |            |             | 0.504          | 0.095           | 67                         | 44,300                        |                      | 3.90         |             |
| 5            |            |             | 0.505          | 0.098           | 63                         | 43,100                        | 39,500               | 3.77         | 3.68        |

FLEXURAL DATA FOR DIBROMOBUTENE DIOL (DBBD) AND  
2,4-TOLYLENE-DIISOCYANATE (TDI),  
TESTED AT 200°F AFTER EXPOSURE TO 200°F FOR 1/2 HOUR

| Spec.<br>No. | Moles |      | Width<br>(in.) | Thick.<br>(in.) | Ultimate<br>Load<br>(lbs.) | Flexural<br>Strength<br>(psi) | Avg.   | E x 10 <sup>-6</sup> |      |
|--------------|-------|------|----------------|-----------------|----------------------------|-------------------------------|--------|----------------------|------|
|              | TDI   | DBBD |                |                 |                            |                               |        | (psi)                | Avg. |
| 1            | 1.0   | 1.0  | 0.500          | 0.097           | 87                         | 55,500                        |        | 3.85                 |      |
| 2            |       |      | 0.497          | 0.097           | 84                         | 54,000                        |        | 3.90                 |      |
| 3            |       |      | 0.514          | 0.098           | 79                         | 48,100                        |        | 3.64                 |      |
| 4            |       |      | 0.499          | 0.096           | 94                         | 58,900                        |        | 4.03                 |      |
| 5            |       |      | 0.489          | 0.097           | 87                         | 56,700                        | 54,600 | 4.08                 | 3.90 |
| 1            | 1.1   | 1.0  | 0.518          | 0.094           | 112                        | 69,000                        |        | 4.14                 |      |
| 2            |       |      | 0.514          | 0.095           | 104                        | 67,400                        |        | 4.14                 |      |
| 3            |       |      | 0.525          | 0.098           | 117                        | 69,600                        |        | 3.64                 |      |
| 4            |       |      | 0.528          | 0.097           | 121                        | 73,900                        |        | 4.11                 |      |
| 5            |       |      | 0.510          | 0.098           | 116                        | 71,000                        | 70,200 | 3.90                 | 3.99 |
| 1            | 1.2   | 1.0  | 0.519          | 0.099           | 108                        | 63,600                        |        | 3.77                 |      |
| 2            |       |      | 0.503          | 0.099           | 93                         | 56,600                        |        | 3.95                 |      |
| 3            |       |      | 0.517          | 0.096           | 102                        | 64,300                        |        | 3.80                 |      |
| 4            |       |      | 0.523          | 0.097           | 103                        | 62,800                        |        | 3.92                 |      |
| 5            |       |      | 0.530          | 0.102           | 113                        | 61,500                        | 61,800 | 3.35                 | 3.76 |
| 1            | 1.3   | 1.0  | 0.500          | 0.100           | 59                         | 35,400                        |        | 3.97                 |      |
| 2            |       |      | 0.495          | 0.099           | 62                         | 38,400                        |        | 3.85                 |      |
| 3            |       |      | 0.498          | 0.095           | 78                         | 52,200                        |        | 4.32                 |      |
| 4            |       |      | 0.504          | 0.095           | 77                         | 50,900                        |        | 3.97                 |      |
| 5            |       |      | 0.518          | 0.098           | 78                         | 47,100                        | 44,700 | 3.70                 | 3.96 |

FLEXURAL DATA FOR HEXAFLUOROPENTANE DIOL (HFPD), MACCONATE 200 AND  
2,4-TOLYLENE-DIISOCYANATE (TDI) TESTED AT ROOM TEMPERATURE

| Spec.<br>No. | Moles |      |      | Width<br>(in.) | Thick.<br>(in.) | Ultimate<br>Load<br>(lbs.) | Flexural<br>Strength<br>(psi) | E x 10 <sup>-6</sup> |       |
|--------------|-------|------|------|----------------|-----------------|----------------------------|-------------------------------|----------------------|-------|
|              | TDI   | HFPD | 200  |                |                 |                            |                               | Avg.                 | (psi) |
| 1            | 1.0   | 1.5  | 0.5  | 0.532          | 0.073           | 42                         | 44,400                        |                      | 3.97  |
| 2            |       |      |      | 0.530          | 0.075           | 43                         | 43,300                        |                      | 3.70  |
| 3            |       |      |      | 0.533          | 0.073           | 38                         | 40,100                        |                      | 3.80  |
| 4            |       |      |      | 0.536          | 0.075           | 42                         | 41,700                        |                      | 3.55  |
| 5            |       |      |      | 0.532          | 0.072           | 38                         | 37,100                        | 41,300               | 4.03  |
|              |       |      |      |                |                 |                            |                               |                      | 3.81  |
| 1            | 0.75  | 1.5  | 0.75 | 0.507          | 0.085           | 63                         | 51,800                        |                      | 4.41  |
| 2            |       |      |      | 0.513          | 0.085           | 65                         | 52,800                        |                      | 4.05  |
| 3            |       |      |      | 0.513          | 0.086           | 72                         | 56,800                        |                      | 3.97  |
| 4            |       |      |      | 0.519          | 0.084           | 66                         | 57,900                        |                      | 4.23  |
| 5            |       |      |      | 0.514          | 0.085           | 62                         | 50,300                        | 53,900               | 4.00  |
|              |       |      |      |                |                 |                            |                               |                      | 4.13  |
| 1            | 0.5   | 1.5  | 1.0  | 0.473          | 0.087           | 50                         | 41,900                        |                      | 3.68  |
| 2            |       |      |      | 0.472          | 0.087           | 53                         | 44,500                        |                      | 3.73  |
| 3            |       |      |      | 0.471          | 0.089           | 51                         | 41,000                        |                      | 3.69  |
| 4            |       |      |      | 0.474          | 0.090           | 53                         | 41,400                        |                      | 3.77  |
| 5            |       |      |      | 0.471          | 0.087           | 50                         | 42,100                        | 42,200               | 3.62  |
|              |       |      |      |                |                 |                            |                               |                      | 3.70  |

FLEXURAL DATA FOR HEXAFLUOROPENTANE DIOL (HFPD), MACCONATE 200 AND  
2,4-TOLYLENE-DIISOCYANATE TESTED AT 200°F AFTER EXPOSURE TO 200°F FOR 1/2 HOUR

| Spec.<br>No. | Moles |     |      | Width<br>(in.) | Thick.<br>(in.) | Ultimate<br>Load<br>(lbs.) | Flexural<br>Strength<br>(psi) | E x 10 <sup>-6</sup> |       |
|--------------|-------|-----|------|----------------|-----------------|----------------------------|-------------------------------|----------------------|-------|
|              | TDI   |     | 200  |                |                 |                            |                               | Avg.                 | (psi) |
| 1            | 1.0   | 1.5 | 0.5  | 0.539          | 0.075           | 7                          | 6,900                         | --No Results--       |       |
| 2            |       |     |      | 0.536          | 0.072           | 8                          | 8,600                         |                      |       |
| 3            |       |     |      | 0.533          | 0.075           | 7                          | 7,000                         |                      |       |
| 4            |       |     |      | 0.532          | 0.071           | 6                          | 6,700                         |                      |       |
| 5            |       |     |      | 0.526          | 0.077           | 7                          | 6,700                         | 7,200                |       |
| 1            | 0.75  | 1.5 | 0.75 | 0.485          | 0.084           | 14                         | 12,300                        | --No Results--       |       |
| 2            |       |     |      | 0.519          | 0.085           | 22                         | 17,600                        |                      |       |
| 3            |       |     |      | 0.515          | 0.086           | 17                         | 13,400                        |                      |       |
| 4            |       |     |      | 0.509          | 0.084           | 21                         | 17,500                        |                      |       |
| 5            |       |     |      | 0.517          | 0.085           | 21                         | 16,800                        | 15,500               |       |
| 1            | 0.50  | 1.5 | 1.0  | 0.473          | 0.089           | 9                          | 7,200                         | --No Results--       |       |
| 2            |       |     |      | 0.471          | 0.096           | 12                         | 9,400                         |                      |       |
| 3            |       |     |      | 0.470          | 0.089           | 14                         | 11,300                        |                      |       |
| 4            |       |     |      | 0.470          | 0.087           | 15                         | 12,600                        |                      |       |
| 5            |       |     |      | 0.470          | 0.087           | 15                         | 12,600                        | 10,600               |       |

FLEXURAL DATA FOR DIBROMOBUTENE DIOL (DBBD), MACCONATE 200, AND  
2,4-TOLYLENE-DIISOCYANATE (TDI) TESTED AT ROOM TEMPERATURE

| Spec.<br>No. | Moles |      |      | Width<br>(in.) | Thick.<br>(in.) | Ultimate<br>Load<br>(lbs.) | Flexural<br>Strength<br>(psi) | E x 10 <sup>-6</sup> |       |      |
|--------------|-------|------|------|----------------|-----------------|----------------------------|-------------------------------|----------------------|-------|------|
|              | TDI   | DBBD | 200  |                |                 |                            |                               | Avg.                 | (psi) | Avg. |
| 1            | 1.0   | 1.5  | 0.5  | 0.468          | 0.096           | 80                         | 55,700                        |                      | 3.55  |      |
| 2            |       |      |      | 0.468          | 0.096           | 78                         | 54,300                        |                      | 4.01  |      |
| 3            |       |      |      | 0.465          | 0.097           | 73                         | 50,000                        |                      | 3.57  |      |
| 4            |       |      |      | 0.469          | 0.096           | 83                         | 57,600                        |                      | 3.62  |      |
| 5            |       |      |      | 0.465          | 0.096           | 80                         | 55,900                        | 54,700               | 3.64  | 3.68 |
| 1            | 0.75  | 1.5  | 0.75 | 0.475          | 0.093           | 55                         | 40,100                        |                      | 3.72  |      |
| 2            |       |      |      | 0.485          | 0.095           | 53                         | 36,400                        |                      | 3.69  |      |
| 3            |       |      |      | 0.487          | 0.096           | 58                         | 38,800                        |                      | 3.55  |      |
| 4            |       |      |      | 0.470          | 0.093           | 57                         | 42,000                        |                      | 4.06  |      |
| 5            |       |      |      | 0.477          | 0.093           | 57                         | 41,400                        | 39,700               | 3.79  | 3.76 |
| 1            | 0.5   | 1.5  | 1.0  | 0.508          | 0.096           | 66                         | 42,300                        |                      | 3.60  |      |
| 2            |       |      |      | 0.508          | 0.097           | 65                         | 40,800                        |                      | 3.76  |      |
| 3            |       |      |      | 0.509          | 0.095           | 65                         | 42,500                        |                      | 3.71  |      |
| 4            |       |      |      | 0.516          | 0.088           | 60                         | 45,100                        |                      | 4.30  |      |
| 5            |       |      |      | 0.509          | 0.099           | 66                         | 39,700                        | 42,100               | 4.70  | 4.01 |

FLEXURAL DATA FOR DIBROMOBUTENE DIOL (DBBD), MACCONATE 200 AND  
2,4-TOLYLENE-DIISOCYANATE TESTED AT 200°F  
AFTER EXPOSURE TO 200°F FOR 1/2 HOUR

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| Spec.<br>No. | Moles |      |      | Width<br>(in.) | Thick.<br>(in.) | Ultimate<br>Load<br>(lbs.) | Flexural<br>Strength<br>(psi) | E x 10 <sup>-6</sup> |       |
|--------------|-------|------|------|----------------|-----------------|----------------------------|-------------------------------|----------------------|-------|
|              | TDI   | DBBD | 200  |                |                 |                            |                               | Avg.                 | (psi) |
| 1            | 1.0   | 1.5  | 0.5  | 0.469          | 0.096           | 80                         | 55,600                        |                      | 3.25  |
| 2            |       |      |      | 0.465          | 0.096           | 72                         | 50,400                        |                      | 3.40  |
| 3            |       |      |      | 0.468          | 0.094           | 78                         | 56,500                        |                      | 3.67  |
| 4            |       |      |      | 0.464          | 0.095           | 79                         | 56,600                        |                      | 3.47  |
| 5            |       |      |      | 0.469          | 0.097           | 78                         | 53,100                        | 54,400               | 3.35  |
|              |       |      |      |                |                 |                            |                               |                      | 3.43  |
| 1            | 0.75  | 1.5  | 0.75 | 0.486          | 0.098           | 59                         | 39,500                        |                      | 3.48  |
| 2            |       |      |      | 0.482          | 0.096           | 57                         | 38,500                        |                      | 3.45  |
| 3            |       |      |      | 0.475          | 0.096           | 58                         | 39,700                        |                      | 3.85  |
| 4            |       |      |      | 0.469          | 0.096           | 55                         | 38,200                        |                      | 3.54  |
| 5            |       |      |      | 0.471          | 0.095           | 56                         | 39,500                        | 39,100               | 3.72  |
|              |       |      |      |                |                 |                            |                               |                      | 3.61  |
| 1            | 0.5   | 1.5  | 1.0  | 0.513          | 0.095           | 58                         | 37,600                        |                      | 3.20  |
| 2            |       |      |      | 0.516          | 0.094           | 57                         | 37,500                        |                      | 3.50  |
| 3            |       |      |      | 0.505          | 0.098           | 60                         | 46,800                        |                      | 3.21  |
| 4            |       |      |      | 0.508          | 0.098           | 58                         | 35,600                        |                      | 3.06  |
| 5            |       |      |      | 0.511          | 0.097           | 54                         | 33,700                        | 38,400               | 3.47  |
|              |       |      |      |                |                 |                            |                               |                      | 3.29  |

FLEXURAL DATA FOR DIBROMOBUTENE DIOL (DBBD), MACCONATE 300, AND  
2,4-TOLYLENE-DIISOCYANATE TESTED AT ROOM TEMPERATURE

| Spec.<br>No. | Moles      |             |            | Width<br>(in.) | Thick.<br>(in.) | Ultimate<br>Load<br>(lbs.) | Flexural<br>Strength<br>(psi) | E x 10 <sup>-6</sup> |             |
|--------------|------------|-------------|------------|----------------|-----------------|----------------------------|-------------------------------|----------------------|-------------|
|              | <u>TDI</u> | <u>DBBD</u> | <u>300</u> |                |                 |                            |                               | <u>Avg.</u>          | <u>Avg.</u> |
| 1            | 1.0        | 1.5         | 0.5        | 0.464          | 0.105           | 156                        | 91,800                        | 4.18                 |             |
| 2            |            |             |            | 0.465          | 0.106           | 149                        | 85,800                        | 3.96                 |             |
| 3            |            |             |            | 0.465          | 0.106           | 161                        | 92,700                        | 3.69                 |             |
| 4            |            |             |            | 0.457          | 0.106           | 155                        | 90,800                        | 3.61                 |             |
| 5            |            |             |            | 0.450          | 0.106           | 158                        | 94,000                        | 91,000               | 3.67        |
|              |            |             |            |                |                 |                            |                               |                      | 3.82        |

TESTED AT 200°F AFTER EXPOSURE TO 200°F FOR 1/2 HOUR

|   |     |     |     |       |       |     |        |        |      |
|---|-----|-----|-----|-------|-------|-----|--------|--------|------|
| 1 | 1.0 | 1.5 | 0.5 | 0.470 | 0.105 | 110 | 63,800 | 3.09   |      |
| 2 |     |     |     | 0.458 | 0.107 | 103 | 59,200 | 3.09   |      |
| 3 |     |     |     | 0.468 | 0.105 | 117 | 68,155 | 3.40   |      |
| 4 |     |     |     | 0.463 | 0.106 | 110 | 63,600 | 3.53   |      |
| 5 |     |     |     | 0.454 | 0.106 | 126 | 70,900 | 65,100 | 3.41 |
|   |     |     |     |       |       |     |        |        | 3.36 |















